

# APPLICATION UNDER UNITED STATES PATENT LAWS

Atty. Dkt. No. PW 0278094  
(M#)

Invention: CONNECTION WITH A COMPONENT PRODUCED OF A THERMOPLASTIC ELASTOMER

Inventor (s): AVIDES MOREIRA

Pillsbury Winthrop LLP  
Intellectual Property Group  
1100 New York Avenue, NW  
Ninth Floor  
Washington, DC 20005-3918  
Attorneys  
Telephone: (202) 861-3000

## This is a:

- ☐ Provisional Application
- ☐ Regular Utility Application
- ☒ Continuing Application  
☒ The contents of the parent are incorporated by reference
- ☐ PCT National Phase Application
- ☐ Design Application
- ☐ Reissue Application
- ☐ Plant Application
- ☐ Substitute Specification  
Sub. Spec Filed \_\_\_\_\_  
in App. No. \_\_\_\_\_ /
- ☐ Marked up Specification re  
Sub. Spec. filed \_\_\_\_\_  
In App. No. \_\_\_\_\_ /

## SPECIFICATION

CONNECTION WITH A COMPONENT PRODUCED OF A  
THERMOPLASTIC ELASTOMER

5

The invention relates to a component produced of a thermoplastic elastomer which is applied in a permanent connection between at least two components in an object and a process for the fashioning of this connection. The invention is particularly aimed at a process for bringing about a sealing connection between two components in an object.

Sealing components of thermoplastic elastomer materials are known and find wide application. For example as a body plug, that is, a plug for sealing holes needed in the construction of, for example, a car body or refrigerator to prevent ingress of water, gasket rings, sealing rings, shrink-on sleeving and the like. Application of sealing objects of thermoplastic elastomer material presents important advantages in that the thermoplastic properties allow more complex mouldings to be mass-produced in a fairly simple manner, which is not possible in the case of rubber materials since, here, vulcanisation needs to take place in the mould. However, a disadvantage of the sealing or connecting objects fabricated of thermoplastic elastomeric material is that they allow only a limited degree of deformation when they are fitted. In general a degree of deformation of the order of some tens of percents is not completely reversible and results in permanent deformation.

If the initial deformation is 100%, permanent deformation often can be as high as 40%.

This is referred to as a tension set of 40%. In general, such tension set is less than 10% for rubbers. In some case, performance is inadequate because of this higher tension set and also because thermoplastic  
5 elastomeric materials generally are harder due to the presence of hard segments in the matrix. Consequently, the object of the invention is to provide a process for the fashioning of a permanent connection between two components of an object, in which at least one  
10 component is obtained by moulding of a thermoplastic elastomeric material, which results in an improved seal.

The object of the invention is achieved by the process described in Claim 1. Thermoplastic  
15 elastomeric materials are described extensively in for example Thermoplastic Elastomers, 2nd edition, G. Holden e.a. editor, Hanser Verlag (1996), ISBN 1-56990-205-4, and the literature cited therein.

The invention can in principle be applied  
20 to all currently common thermoplastic elastomers that are applied in components used in connections. Based on their chemical resistance, for instance, use is preferably be made of thermoplastic elastomers based on polyurethanes, polyesters and polyamides as hard block  
25 in copolymers and combinations of a hard thermoplastic polymer and an elastomer such as polypropylene -EPDM or EPR combinations.

The aforementioned thermoplastic elastomeric materials are commercially obtainable under  
30 various brand names. Blends of hard thermoplastic polymers with thermoplastic elastomers can also be used. The thermoplastic elastomeric materials may contain common additives, for example fillers such as talcum, carbon black, reinforcing fillers such as mica,

stabilizers, colorants and processing aids.

Moulding can be effected using common techniques, for example by injection moulding and extrusion techniques.

5           In general, stretching is effected perpendicularly to the plane in which the connection is to be brought about.

          The degree of stretching needed for the process of the invention is determined from case to case and is much dependent on the shape of the  
10           component and the connection to be applied.

          Stretching here means such amount of deformation as causes more than 10% of the deformation to remain on release of the tension by which the  
15           deformation was brought about. This is normally related to the temperature range from -40°C to +60°C, henceforth referred to as ambient temperature.

          In general, stretching occurs when deformation amounts to more than 50%, and stretching  
20           occurs sooner in harder thermoplastic elastomers than in softer ones.

          In general, stretching will not be applied in excess of 200%, preferably 100%, because otherwise the deformation remaining on release of the deforming  
25           tension, also known as tension set, is too large and less reproducible.

          The time during which stretching is applied may vary between wide limits; for reasons of production it is chosen to be as short as possible, for example  
30           seconds. The required time is determined in part by the reproducibility of the set remaining after relaxation of the component at ambient temperature.

          For reasons of economics, the increased temperature to which the connection eventually is

exposed is chosen to be as high as possible but not higher than a temperature which is about 20°C below the melting point of the thermoplastic elastomer. The melting point of the thermoplastic elastomer is here defined as the temperature at which the summit of the peak in the heating curve of the differential scanning calorimetric (DSC) measurement, at a heating rate of 20°C/min., is located.

It is highly surprising that, under these conditions, the component, having relaxed at ambient temperature, exhibits substantial shrinkage towards the deformation, as a result of which a continuous connection in tension is established.

The time during which the connection is exposed to the increased temperature may vary between wide limits and is often determined by production-related considerations. In general, however, the time should be limited because otherwise the tension at which the connection is brought about will diminish too strongly. A duration of between 0.5 and 30 minutes suffices in most cases.

The invention is illustrated by the following examples.

#### Example 1

In this example the process of the invention is illustrated with reference to a body plug used for sealing holes in a metal sheet.

The body plug is obtained by injection moulding whereupon it is stretched perpendicularly to the plane in which the connection is to be brought about, which, in this example, is effected immediately after the colling of the mould of the injection moulding machine with an attachment in the mold. Next, the stretched

body plug can relax, at ambient temperature of 23°C, to substantially reassume its original dimensions. Once the body plug is inserted in the hole in the metal sheet, the heat treatment at elevated temperature  
5 (170°C) is carried out. In the automotive industry this operation preferably coincides with the paint spraying line. During the latter heat treatment the plug tightly contacts the surface of the metal sheet and is retained in tension.

10 Of course, the dimensions of the body plug are accurately adjusted to suit the deformations to be undergone, and can be established through experiment using test bars of the thermoplastic elastomeric material used.

15 For applications in the automotive industry it is preferred to use polyether ester elastomers based on polybutylene terephthalate as the hard segment inasmuch as, inter alia, such materials are well able to withstand the high temperature occurring in the  
20 paint spraying line and are well paintable.

#### Example II

This example indicates in broad terms the characteristics of the thermoplastic elastomeric  
25 material that need to be determined for proper shaping of the component to be used in the process of the invention and for establishing the suitability of a material for the process of the composition.

Test bars shaped like dumbbell sets with  
30 critical dimensions of 4x20 mm and 1 or 2 mm thick were cut from injection-moulded sheets. Tensile tests were conducted using a Zwick 1445 tensile testing machine. The test bars were deformed at 60°C and the deformation

was maintained for 10 seconds.

After deformation at 60°C the test bars were stored at room temperature for 24 hours with tension released and recovery of the original shape was

5 monitored.

Deformation and recovery of the original shape (relaxation) were measured on the basis of the distance between two markings each provided at 10 mm on either side of the centre of the narrow section of the  
10 test bars.

Relaxation was monitored thereafter for 30 minutes in an oven at 170°C. A parallel measurement was conducted to determine the tension in a test bar whose length was kept constant.

15

Table 1 shows for three copolyether esters the recovery after different deformation as a function of time at 170 °C and under the following conditions: a) 50% initial elongation, b) 100% and c) 200%.

Table 1

d\strain	Arnitel PL380			(A)			Arnitel PL380 B			(B)			Arnitel PL580			(C)		
	25%	50%	100%	25%	50%	100%	25%	50%	100%	25%	50%	100%	25%	50%	100%	25%	50%	100%
di (mm) *1	19,82	19,83	19,58	19,68	19,53	19,79	19,33	19,69	19,49	19,60	19,94	19,63	19,65	19,72	19,65	19,61	19,61	19,61
2	19,44	19,46	19,50	19,52	19,53	19,59	19,45	19,60	19,94	19,63	19,94	19,63	19,65	19,72	19,65	19,61	19,61	19,61
3	19,81	19,74	19,84	19,47	19,41	19,65	19,29	19,55	19,74	19,63	19,94	19,63	19,65	19,72	19,65	19,61	19,61	19,61
dR1 (mm)	20,40	20,93	22,82	30,38	19,58	21,32	23,00	20,80	22,02	28,72	40,56	42,49	44,65	46,81	48,97	51,13	53,29	55,45
	20,18	20,70	24,19	29,89	20,05	21,33	22,92	20,81	22,19	29,94	42,49	44,65	46,81	48,97	51,13	53,29	55,45	57,61
	20,49	20,97	24,62	29,84	20,12	21,50	22,71	20,59	22,65	29,67	44,65	46,81	48,97	51,13	53,29	55,45	57,61	59,77
dR2 (mm)	20,22	20,76	21,47	24,18	18,98	20,23	20,55	20,28	20,87	24,80	32,64	34,80	36,96	39,12	41,28	43,44	45,60	47,76
	19,94	20,44	21,79	23,41	19,22	20,12	20,70	20,24	21,24	23,94	33,20	35,40	37,60	39,80	42,00	44,20	46,40	48,60
	20,26	20,75	21,82	24,03	19,38	20,11	20,46	19,85	21,42	24,50	33,46	35,72	37,98	40,24	42,50	44,76	47,02	49,28

\* Number of the sample

- 5 Where **di** is the initial distance between two marks, **dR1** is the distance between the two marks after relaxation at 23°C and **dR2** is the distance between the two marks after relaxation at 170°C.



The data in the table indicate that polyether ester B seems most suited for the process of the invention because recovery of the original  
5 dimensions after shrinkage at 170°C is high and the tension remains at a high and constant level especially when the initial deformation has been 100% or more.

The latter is also the case with copolyether ester C; however, the residual deformation  
10 remains high. In addition, the hardness of C is higher than that of B.

A = Arnitel PL380®, Shore D hardness = 38  
B = Arnitel PL380® B, Shore D hardness = 38  
15 modified form of Arnitel PL380®  
C = Arnitel PL580®, Shore D hardness = 58  
Arnitel P® copolyether ester based on polybutylene terephthalate as hard segment and polypropylene oxide endcapped with ethylene oxide as soft segment, a  
20 commercial product of DSM, of the Netherlands.